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IONIC LIQUID LUBRICATION OF MEMS DEVICES: AN AFM BASED ANALYSIS AND EVALUATION ON TEST DEVICES (Preprint)

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14. ABSTRACT

Lubrication of Micro Electro Mechanical Systems (MEMS) became very important as the devices became complex and more and more parts had interacting areas. In previous reports, self-assembled monolayers and long chains of flurocarbons were used to lubricate MEMS components with significant success. In this report, a method based on atomic force microscopy is described that measures and compares ionic liquid lubricity. Effect of ring structure is studied in the case of substituted pyridinium and imidazolium rings as cations in ethyl methyl pyridinium and ethyl methyl imidazolium ethyl sulfate. Effect of alkyl chain length on friction was studied for butyl methyl pyrrolidimium and hexyl methyl pyrrolidinium bis(trifluro methyl sulfonyl) imide. Some of the ionic liquids that exhibited promising results from AFM study are tested on MEMS test devices. The friction and wear data obtained for these liquids applied on hydrogenated silicon showed ample correlation to the failure life span of hydrogenated MEMS test devices. This shows that AFM-liquid cell based tests of ionic liquid lubricity is a good characterization technique for screening lubricants for MEMS devices.

15. SUBJECT TERMS
Ionic liquid, atomic force microscopy, boundary lubrication, MEMS

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Ionic liquid lubrication of MEMS devices: An AFM based analysis and evaluation on test devices.

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Abstract

Lubrication of Micro electro mechanical system (MEMS) devices became very important as the devices became complex and more and more parts had interacting areas. In previous reports self assembled monolayers and long chains of flurocarbons were used to lubricate MEMS components with significant success. In this report, a method based on Atomic Force microscope is described that measures and compare ionic liquid lubricity. Selected ionic liquid molecules from AFM study are used to control friction and wear of MEMS test devices. Effect of ring structure is studied in the case of substituted pyridinium and imidazolium rings as cations in ethyl methyl pyridinium and ethyl methyl imidazolium ethyl sulfate. Effect of alkane chain length on friction was studied for butyl methyl pyrrolidinium and hexyl methyl pyrrolidinium bis(trifluro methyl sulfonyl) imide. The friction and wear data obtained for these liquids applied on hydrogenated silicon showed ample correlation to the failure life span of hydrogenated MEMS test devices lubricated with same liquids. This shows that AFM –liquid cell based tests of ionic liquid lubricity is a good characterization technique for screening lubricants for MEMS devices.

Introduction

Micro electro mechanical system devices find their way into many useful applications. Most of these devices depend on the impact contact interfaces of MEMS. Very few devices have come into market form sliding contact type MEMS. One of the major reasons for the lack of sliding contact devices is the lack of proper lubrication schemes for these devices. In MEMS devices, due to their large surface area to mass ratio, surface forces rather than inertial forces, determine tribological behavior of interacting surfaces. Surface wear and stiction are the significant failure causes of MEMS devices (1-4) To extend the wear life of these devices, a number of engineering solutions were applied before. The roughning of interacting surfaces provided a reduced contact area. Low surface energy films coated on surfaces provide better solution for the reduction of friction and wear thus extending working life. These include self assembled mono-layers, PFPE chains with different functional group terminations and solid lube like Diamond like carbon (5-11). However, large aspect ratio of interacting surface structures hindered these coatings to be uniform or conformal on these surfaces reducing their effect in extending the failure life. Self-assembled monolayers (SAMS) with various backbone chain lengths and functional groups were apllied by different authors. Alkane based coatings provided anti-stiction capabilities that are comparable to that of silanes (12,13). A comparative study of the effectiveness of chain length in controlling the nano scale friction has been studied by a number of groups. A decrease in friction with increased

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chain length has been generally observed from all these studies. (14-17). Behavior of fluerocarbon chains (Z-dol) with bound and mobile phases were studied by applying it on MEMS test devices. Addition of the mobile phase dramatically improved the lubrication performance this material system (18). Bound monolayers of 1-decanol and 1, 7 heptanediol combined with a mobile layer of pentaerithritol ester gave better failure life for test machines (19).

All the above mentioned techniques could substantially increase the wear life of MEMS test machines. However, uniformity and statistical life spread are required to achieve a higher level of satisfaction for all these coating schemes. Average failure life extension and a reduction in the variance for the life are the desirable qualities of a coating scheme. Another interesting aspect of these coatings is the effect of shelf life on wear life. Since these devices are not readily used for their purposes, the storage times for these devices need to be considered in evaluating their failure life. The tests made on aged coated machines suggested that more reliable coating schemes might be needed to sustain the wear life of aged machines.

In this work, room temperature ionic liquids (RTILs) are considered as lubricating materials for MEMS test devices. Since there are a number of choices of RTILs that can be selected for this purpose and the absence of any particular structural or chemical attributes that we can coordinate to the lubricating capability of these molecules, development of an easy method for the comparison of lubrication performance of ionic liquids would be highly desirable. The purpose of this study is to develop such a method

and applying it in selecting certain ILs that have potential to be lubricants. There are four RTILs slected based on certain physical qualities that may give rise to low dynamic friction for low normal load interfaces. Pyridinium and imidazolium rings are compared as cations in ethyl methyl pyridinium (EMPy-ES) and ethyl methyl imidazolium ethyl sulfate (EMI-ES) respectively, which are low melting ($\sim$ -60 $^\circ$ C) and stable under humidity. It has been observed that certain organic materials with relatively long alkyl chains give rise to low friction in micro-scale measurements [16]. The effect of alkyl chains length is reported for butyl methyl pyrrolidinium bis-(trifluromethyl sulfonyl) imide (BMPr-Imide) and hexyl methyl pyrrolidinium bis-(trifluromethyl sulfonyl) imide (HMPr-Imide.) in this work.

The tribo testing method developed for the lubrication comparison of these ILs are based on conventional AFM attached with a fluid cell with standard cantilever replaced with one attached with a micro-sphere. This set up is selected here to avoid the uncertainties related to single asperity contacts like contact area, local surface roughness variation and flow conditions dissimilarity of lubricating films. Since interface is formed with a sphere of known radius on a disk, this constitutes a standard configuration of contact and multiple asperity contact accomplished would statistically average the variations in the contacts and would give a mean value for the lubrication performance of the tested IL.

The friction force measured under these liquids is a good indication of lubrication potential of them for MEMS interfaces. As observed by others, longer alkyl chains gave lower friction compared to shorter alkyl chains. The six member ring structure was

comparatively more effective in lowering the friction than five member ring structures. Hydrogenation of silicon surface significantly lowered the friction as reported earlier by others (20).

Experiment

The comparison of lubrication performance of selected ionic liquids is made using a conventional atomic force microscope (AFM). The lateral force measurements are made under ionic liquids using a liquid cell arrangement as shown in Figure 1. Interface is formed using 12 miro-meter diameter borosilicate ball and polished cleaned single crystal silicon of two different surface finishes, neat or hydrogenated. Hydrogenation is accomplished by immersing in 53% HF for two min. and subsequent rinsing in two separate baths of methanol for five min. each. The normal load applied varied from 70 to 490 nN. The scanning speed was varied from 5 to 55 micro-meters/s for each normal load selected while the scanning length remained at 3 micro-meters. The lateral force Lf caused the torsional displacement of the cantilever and can be calculated as

$$Lf = (V_{trace} - V_{re\text{-}trace})/2 \ K_{lat} \ S_{lat}.$$

where V_{trace} and $V_{retrace}$, are the potential generated on photo detector, which can be obtained from friction loop (see inset of Figure 1) recorded in each friction scan as the average of 256 values (number of pixels in each scan line) measured in each way. They are the resultant electric potentials formed due to displacement of the laser spot from the reference position on position sensitive photo detector. K_{lat} and S_{lat} are the lateral spring

constant of the cantilever and lateral sensitivity of photo diode respectively. Lateral force measured can be expressed as

$$Lf = F1 + F2$$

F1 is the viscous drag on the ball and F2 is the friction originating from interface. F1 can be estimated separately by forcing F2 to zero by measuring Lf with a raised poison of the cantilever. It was estimated that approximately 2-3% of the total lateral force was caused by the viscous drag at low velocities. There was on average 5% variation observed in the total lateral force measurements. The wear scar depth in each case is measured by a sharp tip after replacing the cantilever used for friction measurement. Wear test conditions are as follows: three full scans of a 3x3 μm^2 area, 256 lines of scan per frame scanned in around 6.5 min total time. The normal load and sliding speed for these scans are 140 nN and 6 μm/s respectively.

Surface chemistry was studied with a Surface Instruments (SSI) M-probe XPS instrument operated at a base pressure of 3 x 10^{-7} Pa. Using an A1 anode, a monochrometer, and a 25 eV pass energy, the full width at half maximum (FWHM) of the Au 4f7/2 peak was 0.71 eV. Binding energy positions were calibrated against the Au 4f7/2 peak and the energy separations were calibrated using the Cu 3s and Cu 2p3/2 peaks at 122.39 and 932.47 eV, respectively.

The chemical groups attached to the surfaces were recognized by FTIR techniques.

Nicolet 6700 form Thermo Electron Corporation was used to analyze these samples. The settings chose for the scanning were, Mercury Cadmium Telluride (MCT/A) as detector and Potassium bromide (KBr) for both window and beam-splitter.

Failure life of lubricated and un-lubricated MEMS test machines, (Figure 2) described elsewhere (21) are found out by using an environmentally controlled probe chamber. A triangular wave pattern of 1 kHz and 100V amplitude was used to excite these devices. The surrounding Relative humidity in the probe chamber was kept around 2%. Devices were run continuously until it fails or the slider amplitude was reduced considerably (80%). The time elapsed for the failure was recorded and multiplied by the frequency to calculate failure life in cycles. An average life of 6 - 7 machines is reported as the failure life of the devices.

Sample preparation

MEMS test devices used for the failure life analysis were released and coated with ionic liquids using the following procedure. Two dies were cleaned in acetone for 30 min. and subsequently in methanol for 5min. Structures were released by putting in 52% HF for two and a half min. and rinsing in methanol twice for 5 min. each. They were dried in dry nitrogen flow at room temperature and transferred to 0.1% ionic liquid solution in acetonitrile without much delay and left there for one hour. Took out and washed repeatedly in acetonitrile and methanol for 5 min. each and dried fast in dry nitrogen flow. If majority of the devices were not released at this time washing cycle is repeated

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with acetonitrile and methanol and dried fast in dry nitrogen flow to get more devices released. The samples for FTIR and XPS were prepared on clean single crystal silicon wafers just following the same procedure described above. Samples prepared without hydrogenation (avoiding HF etch) cycle is named neat silicon.

Results and discussion

As described in experimental section the lateral force variation on the cantilever for interface formed between micro-sphere and silicon surface lubricated with each ionic liquid was noted for different applied normal load and scanning speeds. Friction coefficients were calculated form this data taking scanning speed as a parameter. Figure 3(a) and 3(b) show friction coefficient for two selected molecules as a function of scanning speed taking normal load as a parameter for each curve. Figure 3(a) shows friction coefficient for BMPr-Imide and Figure 3(b) shows that for HMPr-Imide. From these figures it can be noted that longer alkyl chain substituted HMPr-Imide provides overall better lubrication. Also we notice that the overall friction is increased substantially (0.3 to 0.6) with normal load for HMPr-Imide at lower scanning speed. This might be attributed to longer chains being squished under heavy load thereby the micro-sphere approaches silicon roughness while in the case of shorter chains the micro-sphere is already in the proximity of silicon. The similar friction coefficient values obtained in these cases substantiate this concept. As the sliding speed increases this effect gets diminished since at higher speed molecules can be partially pushed out of the interface since these molecules are not chemically attached to the surface.

Contact wear generated from friction forces is a good measure of quality of interface

lubrication. Figure 4(a) and 4(b) shows the wear scar resulted from the contact scans of

interfaces lubricated with BMPr-Imide and HMPr-Imide respectively. The sliding

conditions chosen are low load and speed (see experiment section). As evident form the

figures, the wear for BMPr-Imide is much larger (4.6 nm) than that of HMPr-Imide case

(0.8 nm). Lower friction and wear in the case of HMPr-Imide, suggest a screening effect

of longer alkyl chains that are attached to the cations which might be remaining at the

interface for low sliding speeds.

If we consider the hydrogenated silicon instead of neat as one of the interface surfaces for

friction studies it has been shown in earlier studies that friction is significantly lower.

Comparison of the lubrication effect of six member ring to five member ring is carried

out on a hydrogenated silicon surface. Figure 5(a) and 5(b) shows coefficient of friction

generated for interfaces lubricated with EMPy-ES and EMI-ES respectively. As

expected the average friction is much reduced (almost one order of magnitude) compared

to that of neat silicon lubricated interfaces. Between these two ionic liquids we don't

notice a big change in friction coefficient, however in the case of EMI-ES the friction

values are more spread and have slightly higher average compared to those of EMPy-ES.

The highly reduced friction with hydrogenation can be accounted for by considering the

following possible chemical reaction between lubricant and h-silicon.
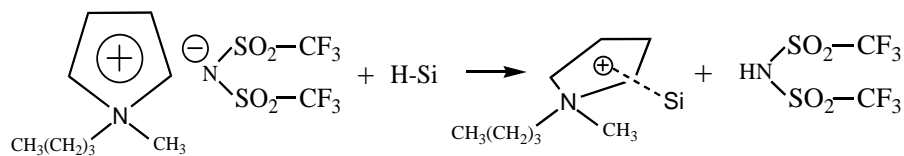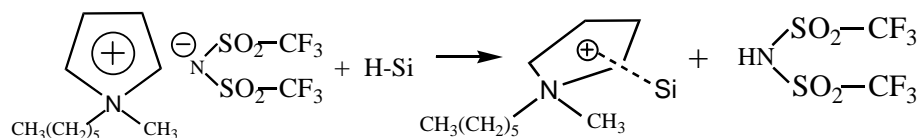
In the above described reaction the cataions of these ionic liquids replaces hydrogen on

the silicon surface and get attached there while the freed hydrogen combines with anaion

and freely moves in the liquid. As a result of the reaction a mono-layer of cataions forms

on the silicon surface and act like a screen between the interfaces. Formation of this

screen might be the reason behind the drastic friction reduction observed in these cases.

A slightly lower average friction observed for EMPy-ES is probably due to the fact that

arranging hexagons (tessellation) on a plane is much easier and complete compared to

pentagons. The screening effect is not diminished even at the higher speed probably

because the screen formed in both cases stays at the interface due to its chemical

attachments.

Formation of the cataion screen and subsequent separation of the freely floating anaion

entity can be further inferred from the results obtained from FTIR and XPS of the H-

silicon surface reacted with corresponding ionic liquids. Figure 6(a) shows the FTIR

spectrum observed for EMPy-ES coated H-silicon superimposed with spectrum obtained

for neat silicon coated with same ionic liquid. As we can see form the figure peaks

expected for Pyridinium ring vibrations are present only for H-silicon substantiating the

fact that cataion attachment remains only for H-silicon surface after the rinsing. The

evidence for the loss of anaion containing sulfur in the rinsing process is obtained if look

for the infrared absorption region for SO stretch peaks. Figure 6(b) shows SO stretch region for EMPy-ES coated H- silicon superimposed with neat EMPy-ES absorption spectrum. Absence of SO strech peaks from EMPy-ES on coated H-silicon implies the loss of the anaion in the process of rinsing due its unattached state. Table 1 gives the XPS analysis data for EMPy-ES coated and rinsed silicon wafer. Absence of any intensity for any sulfur peak also substantiates the loss of sulfur containing anaion from this surface.

Figure 7(a) anf 7(b) shows the similar results obtained for BMPr-Imide used in this study. Similar arguments can be made in this case also to prove the separation of anaion and cataion and subsequent attachment of the cation to the H-silicon and washing off the sulfur containing anaion in the process of rinsing. Table 2 provides the XPS data corresponding to this material. Even though FTIR and XPS provides evidence for the formation of a screen between the interfaces, the actual nature of this bonding is still not fully understood. Data from other analytical techniques need to be collected and studied before we can describe these interactions with complete details.

Just like we observed in the neat silicon case, the wear scar formed in the H-silicon hints the lubrication performance of ionic liquids. Figure 8(a) and (b) shows the topography and corresponding line section of the wear scar obtained in the case of BMPr-Imide. The wear scar is barely seen with line section showing no detectable wear suggesting a very low friction probably due to the screening effect we suggested earlier.

The overall friction behavior of ionic liquid lubricated interfaces studied with liquid cell attached AFM setup is shown in Figure 9. The left most value being that for bare silicon and next one is that for BMPr-Imide, which has comparatively higher friction than others. As we can see different ionic liquid molecular structures gave rise to different coefficient of friction reflecting their lubrication capability. For all the cases studied the hydrogenation of the surface reduced the friction substantially. Longer alkyl chain substituted and six member ring structure based cataions gave more desirable friction values.   The friction observed in the case of HMPr-Imide, EMI-ES and EMPy-ES were so promising (especially H-silicon cases) that they were selected for testing on MEMS test devises. The test devices were coated as described in experiment section. It can be seen that the failure life of these machines are closely connected to the friction coefficients measured by the AFM setup.

Failure life of MEMS test devices lubricated with ionic liquids.

Figure 10 shows the results from lubricated MEMS test machines failure analysis.   The first one from left side shows failure life of an un-lubricated machine followed by a fluorinated ionic liquid taken form a previous study given just for reference purpose. As we can see the un-lubricated machines fail suddenly while coating gives longer life for these machines. An interesting pattern of failure life variation is noted if we combine these results with average friction measured for the corresponding ionic liquids given in Figure 8. As the average friction decreases the average failure life of the machines

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coated with corresponding ionic liquids increases. This co-relation of failure life to average friction gave the testing scheme developed here the advantage of being a suitable method in selecting ionic liquid lubricants in MEMS divices. This test method can be used to characterize low viscous liquid lubricants for their lubrication performance for applications related to low load and low interface conditions.

Conclusion

A method for the study of friction and wear of ILs at *micro-scale* was developed using an AFM liquid cell, which can effectively monitor ionic liquid molecular geometry characteristic influences on the friction coefficient.   Considerable friction and wear reduction was observed for longer alkyl chains substituted cations by studying butyl and hexyl substituted pyrrolidinium cations.   Average friction coefficient was slightly reduced for pyridinium ring compared to imidazolium ring.   An order of magnitude decrease in friction and wear on H-Si surfaces was observed and correlated to an improved bonding of cation structures to H-Si surfaces and a possible mechanism of this bonding was suggested. The findings of AFM based friction study was directly correlated to the failure life of MEMS machines lubricated with ionic liquids.

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Table 1 XPS data for BMPr-Imide coated and rinsed H-Si

XPS line	C1s	O1s	N1s	F1s	S2s	Si2p	Cu2p3
Atom %	18	29	0.2	0.8	0.0	51	0.06

Table 2 XPS data for EMPy-ES coated and rinsed H-Si

XPS line	C1s	O1s	N1s	F1s	S2s	Si2p	Cu2p3
Atom %	19	20	0.2	0.7	0.0	60	0.11

Liquid cell
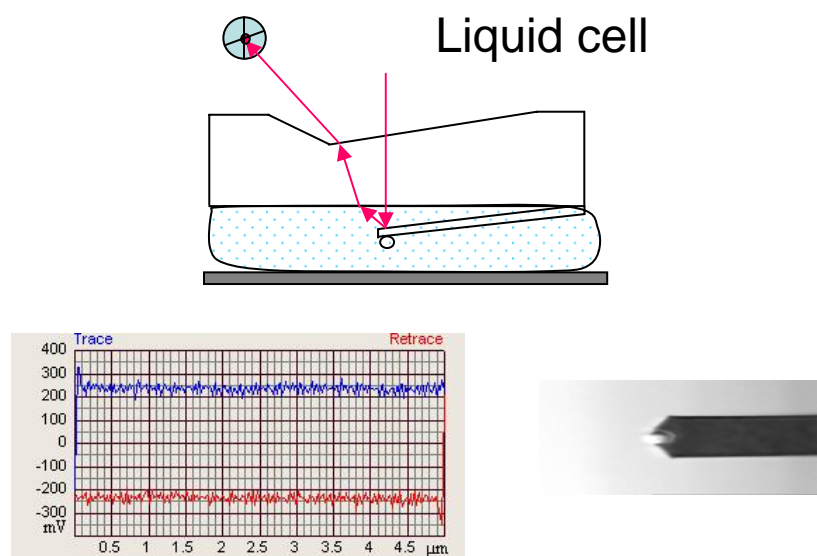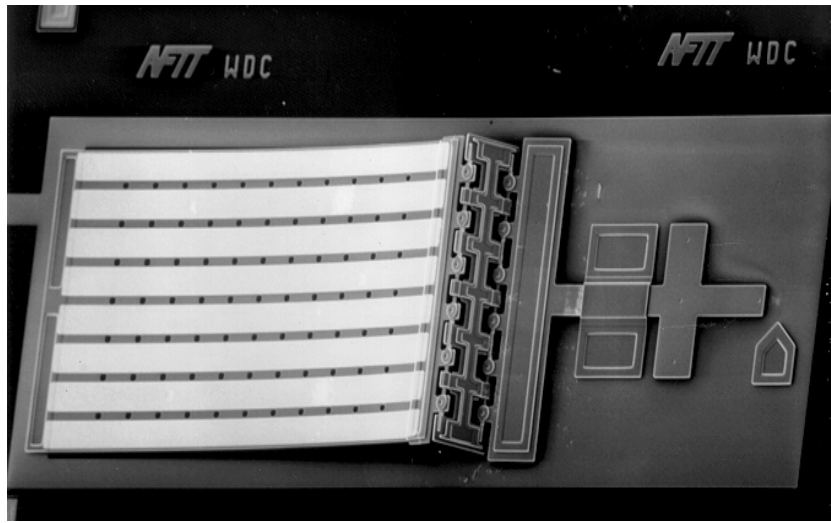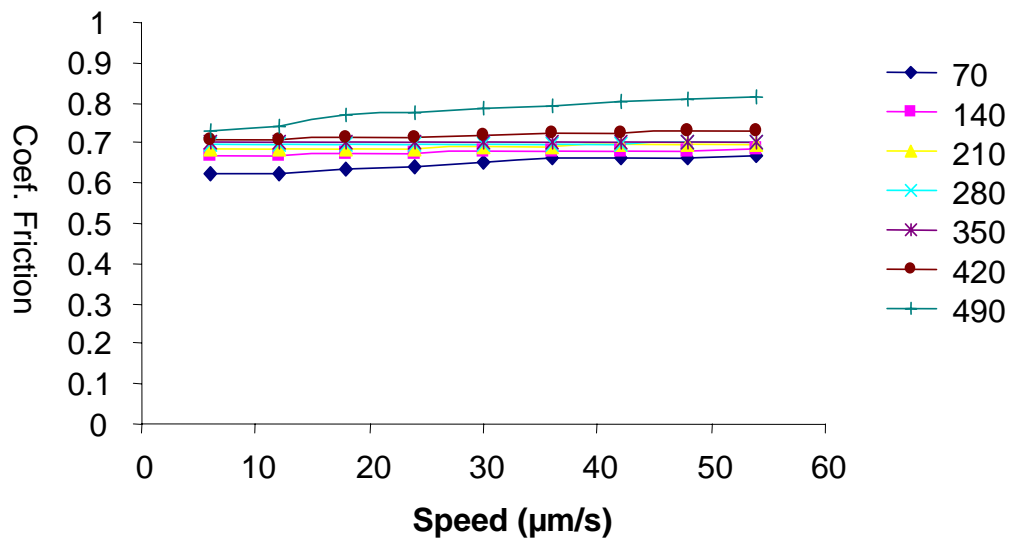
Figure 1.

Figure 2.

Figure 3(a)
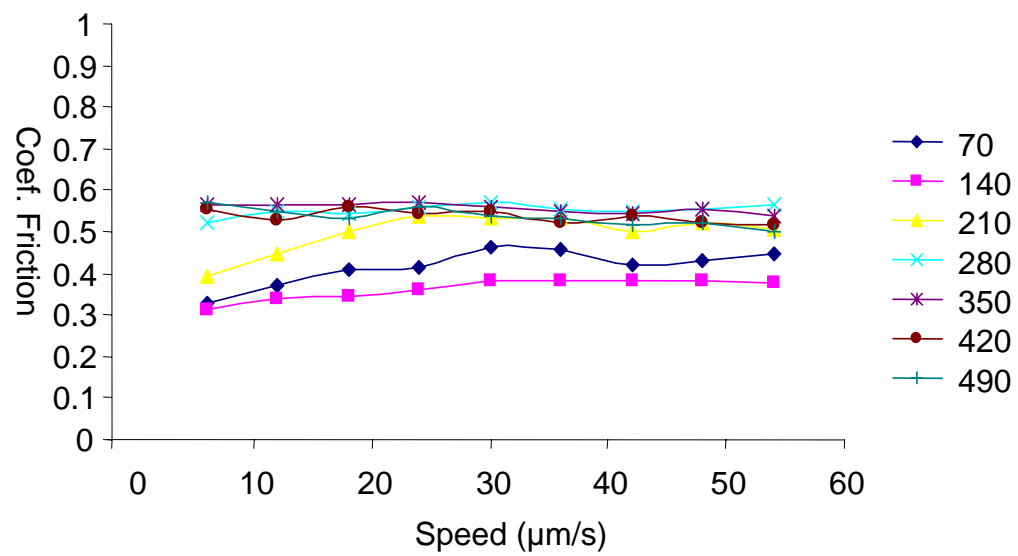
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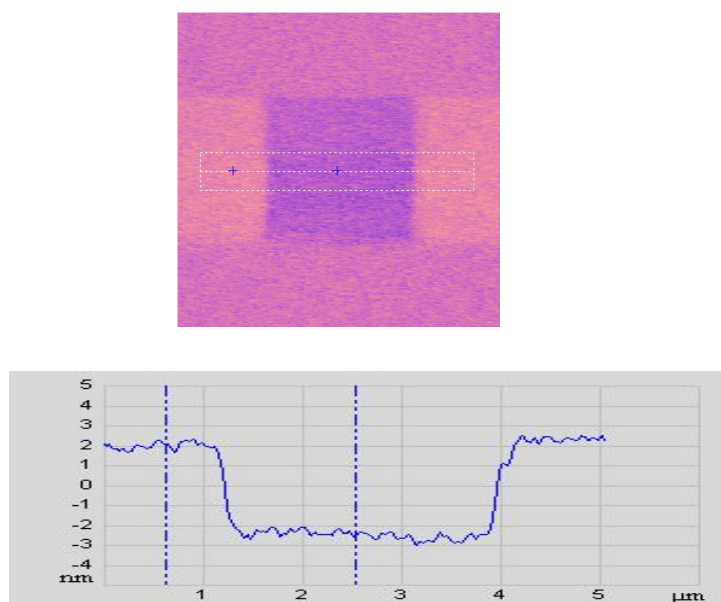
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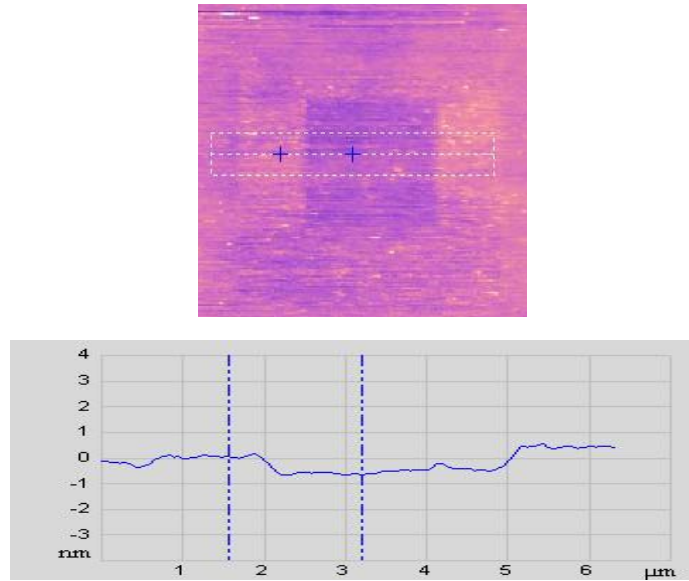
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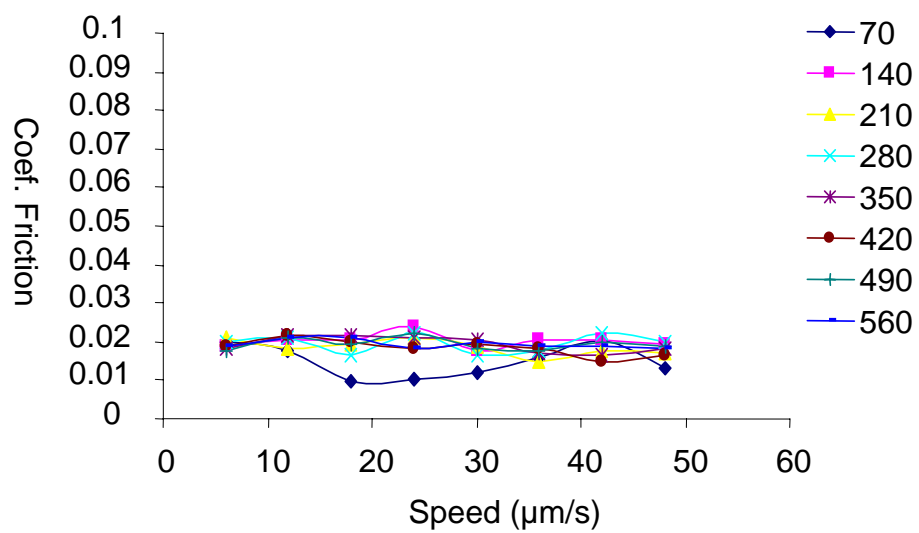
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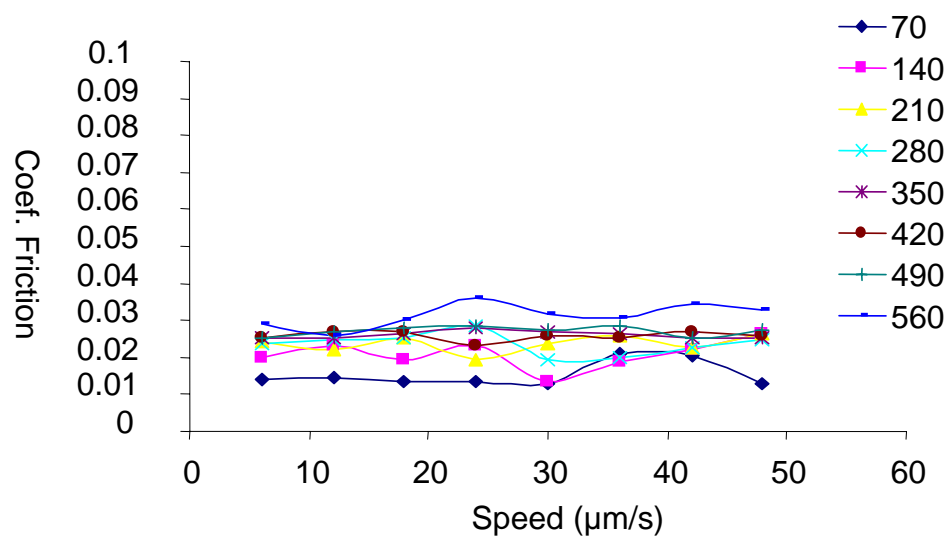
Figure 5(b)

Figure 6(a)

Figure 6(b)

Pyridinium ring region
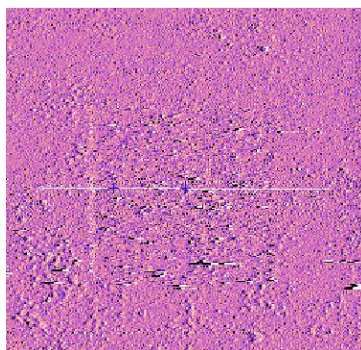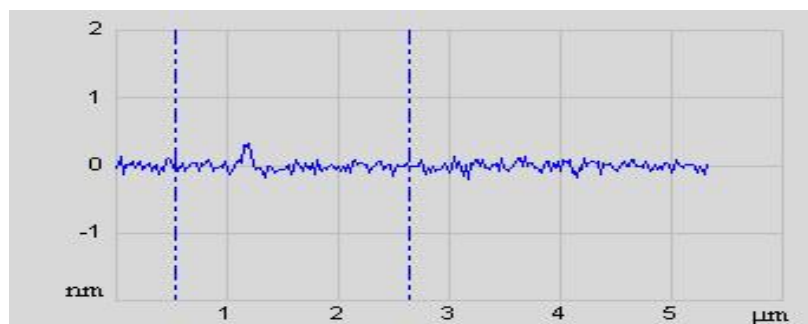
% Transmittance, arb. units, linear scale

Wave numbers (cm-1)
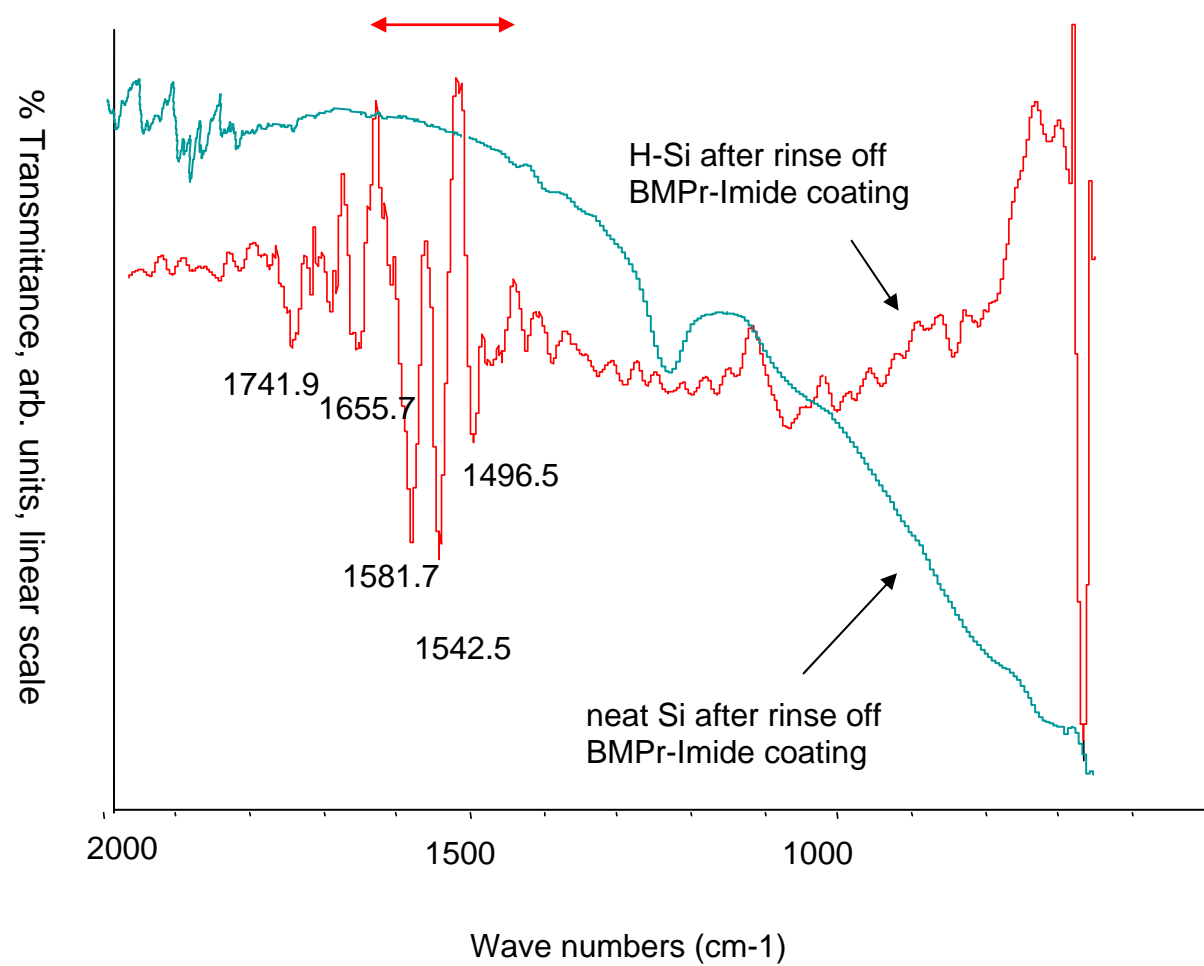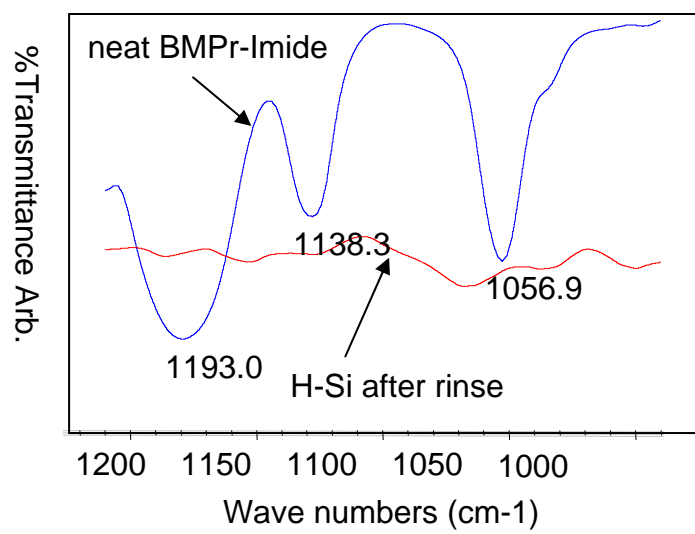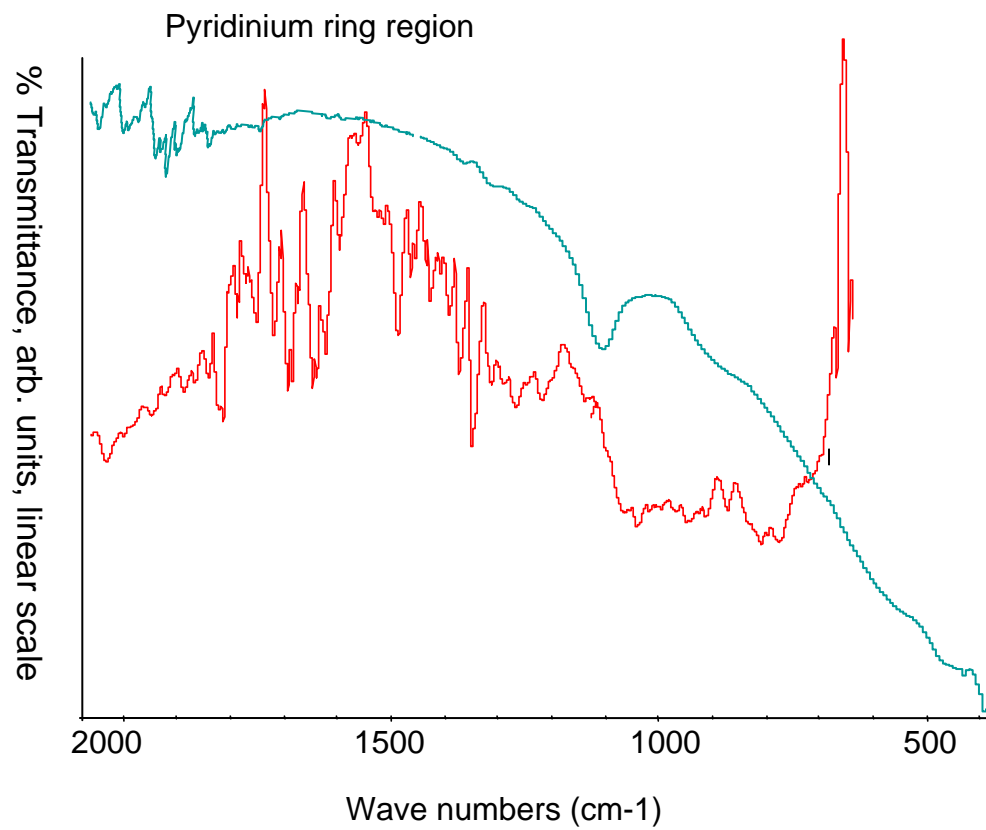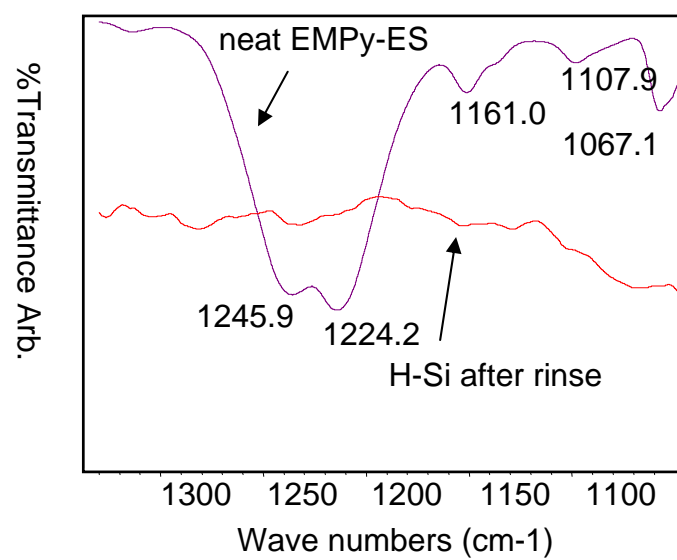
Figure 7(a)

Figure 7(b)

(a)

(b)

Figure 8

Figure 9

Figure 10